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2. Gain on a blue (460.30 nm) transition of singly-charged Xe has been observed in a segmented, linear array of microdischarges, fabricated in a ceramic multilayer structure having an active length of ?1 cm. This is the first example of a microdischarge-driven

optical amplifier.

3. The first large arrays of microdischarges have been fabricated and characterized. Nine hundred (900) pixels have been integrated into 8.5 mm2 of Si, which corresponds to >104 pixels-cm-2. An order of magnitude increase in this packing density should be feasible. The critical innovation (?trick?) to making arrays larger than ??5 x 5 work is the design of the dielectric.

4. Arrays as large as 13 x 13 pixels in ceramic structures have been achieved by individually ballasting each pixel with integrated thick film resistors. Much larger arrays are undoubtedly feasible.

5. Devices having dimensions and volumes as small as (10 um)2 and 10 nL, respectively, have been operated successfully.

6. An array of microdischarges has been shown to significantly decrease the starting voltage for an arc lamp, addressing a major concern of the lighting industry.

7. Eight patent applications have been filed with the U.S. Patent and Trademark Office and the claims for another patent have been granted by the Examiner. A small company started several years ago by two of Dr. Eden's Ph.D. students to commercialize microdischarge technology is (among other things) developing instruments for Arnold AFB (Tennessee) and Tyndall AFB (Florida) under two contracts.

FINAL TECHNICAL REPORT ON AFOSR GRANT No. F49620-00-1-0372

"Microdischarge Devices and Arrays"

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January 2005

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SUMMARY

Under the support of AFOSR grant no. F49620-00-1-0372, we have been pursuing the properties and applications of microdischarge devices having spatial dimensions in the 10-100 μ m range. During this grant, several major advances have been made in the technology of this new family of photonic devices. Highlights include:

- 1. The discovery that microdischarge devices with Si pyramid cathodes are extraordinarily sensitive photodetectors in the near-IR and visible. This new hybrid semiconductor/plasma device was found to have a photoresponsivity at least an order of magnitude greater than that of commercially-available avalanche photodiodes and two orders of magnitude larger than the response of conventional Si photodiodes.
- 2. Gain on a blue (460.30 nm) transition of singly-charged Xe has been observed in a segmented, linear array of microdischarges, fabricated in a ceramic multilayer structure having an active length of ~1 cm. This is the first example of a microdischarge-driven optical amplifier.
- 3. The first large arrays of microdischarges have been fabricated and characterized. Nine hundred (900) pixels have been integrated into 8.5 mm² of Si, which corresponds to >10⁴ pixels-cm⁻². An order of magnitude increase in this packing density should be feasible. The critical innovation ("trick") to making arrays larger than ~5 × 5 work is the design of the dielectric.
- 4. Arrays as large as 13 × 13 pixels in ceramic structures have been achieved by individually ballasting each pixel with integrated thick film resistors. Much larger arrays are undoubtedly feasible.
- 5. Devices having dimensions and volumes as small as $(10 \mu m)^2$ and 10 nL, respectively, have been operated successfully.

- 6. An array of microdischarges has been shown to significantly decrease the starting voltage for an arc lamp, addressing a major concern of the lighting industry.
- 7. Eight patent applications have been filed with the U.S. Patent and Trademark Office and the claims for another patent have been granted by the Examiner. A small company started several years ago by two of Dr. Eden's Ph.D. students to commercialize microdischarge technology is (among other things) developing instruments for Arnold AFB (Tennessee) and Tyndall AFB (Florida) under two contracts.

These are only a few of the accomplishments realized thus far under this AFOSR grant.

Incorporating carbon nanotubes into microdischarge devices, excitation of phosphors with arrays, and the demonstration of readily manufacturable, flexible arrays have also been achieved.

I. MAJOR ACCOMPLISHMENTS UNDER AFOSR GRANT F49620-00-1-0372

A. Microplasma/Semiconductor Photodetectors

We recently observed that microdischarge devices in Si exhibit considerable photosensitivity in the visible and near-infrared (IR). Detailed measurements made over the course of a year with microdischarge devices having inverted square pyramidal cathodes show convincingly that this new hybrid semiconductor/plasma photodetector is, in fact, considerably more sensitive than a Si avalanche photodiode (APD).

Figure 1 shows the spectral response (expressed in A-W⁻¹) for a 100 μ m square pyramidal Si microcavity operating in 400 Torr of Ne. The dielectric consists of SiO₂ and a dry etchable polyimide film (~8 μ m). For comparison, the sensitivity of a commercially-available (Hamamatsu S2381) APD, having a gain up to 100, in this spectral region is also illustrated. Although the spectral profile of the microdischarge response is quite similar to that for the Si APD, the absolute responsivity of the microdischarge exceeds that for the Si device by more than an order of magnitude.

Data similar to those of Fig. 1 but for a $(50 \,\mu\text{m})^2$ microdischarge device are presented in Fig. 2 for a Ne pressure of 800 Torr. Because of its smaller dimensions and the impact of pd scaling, this device prefers to operate at higher pressures. Notice that the peak response for this device (~10³ A/W) is almost 35% higher than that for the larger $(100 \times 100 \,\mu\text{m}^2)$ device and that maximum sensitivity has moved from the near-IR to the red (λ ~620 nm).

Spectral Response

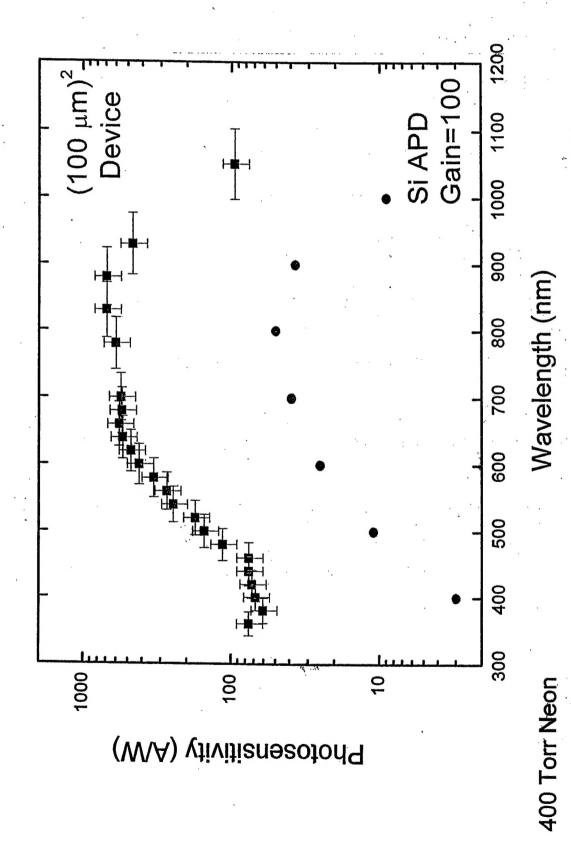


Fig. 1. Comparison of the responsivity of a 100 μm square microdischarge device (p_{Ne} = 400 Torr) with a commercially available APD (manufacturer's specifications).

Spectral Response: $(50 \mu m)^2$

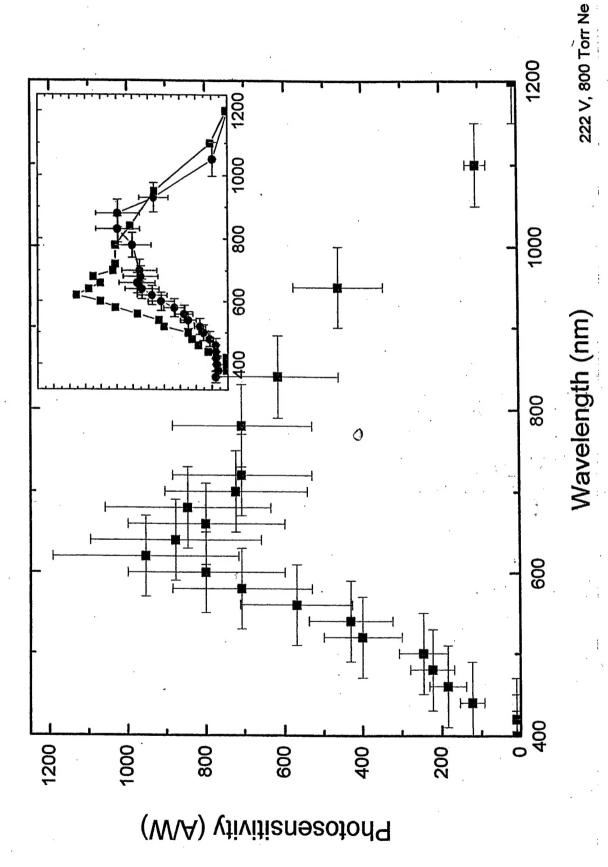


Fig. 2. Data similar to Fig. 1 but for a 50 μ m square device (p_{Ne} = 800 Torr). The inset compares the responses of the $(50 \ \mu m)^2$ and $(100 \ \mu m)^2$ devices.

We interpret the high responsivities of these hybrid semiconductor/plasma devices in terms of the combined action of a photocathode and the plasma — specifically, the effect of enhanced photoelectron emission at the cathode surface in concert with electron avalanche. Preliminary results of numerical simulations of these devices indicate that the electric field strengths at the photocathode surface during normal operation are enormous — 100-400 kV-cm⁻¹. Consequently, it is likely that photogenerated electrons are able to tunnel directly through the vacuum barrier into the plasma. Once free of the semiconductor, photoelectrons are rapidly accelerated under the influence of the strong electric field in the cathode fall and are reproduced by impact ionization and subsequent avalanche.

These considerations and the data of Figs. 1 and 2 strongly suggest that the response of the device is determined primarily by the Si photocathode and the plasma is, to a first approximation, transparent. At very high plasma power loadings (such as that present for the $(50 \ \mu\text{m})^2$ data of Fig. 2), the opacity of the plasma rises and we believe the shift in the wavelength of peak response from the near-IR to the red to be the result of strong Ne transitions in the ~620 nm region. Thus, the optical properties of the plasma can no longer be ignored.

In short, Si microdischarges represent a new and very interesting photodetector concept. The device resembles a photomultiplier except that the conventional dynode chain has been replaced by a low temperature plasma functioning at atmospheric pressure. Since the photoresponse is determined primarily by the photocathode, we are optimistic that the operating range of this device can be extended into the IR and deep-UV with the proper choice of semiconductor photocathode.

B. Large Si Device Arrays

Because of the prospect for integration with electronic devices and subsystems, Si-based microdischarge devices and arrays are of particular interest. Under this AFOSR grant, we have employed photolithographic and conventional wet and dry etching techniques to produce large and precisely defined arrays. Most of the work to date has centered on microdischarge devices having inverted square pyramidal cathodes (or anodes). Developed several years ago, also with AFOSR support, the inverted pyramid devices have proven to be reliable and an exhaustive characterization of their electrical and optical properties over the past 2+ years has resulted in considerable improvements in their lifetimes and output power.

The success of the arrays to be described later was critically-dependent upon the dielectric chosen for the device. Early inverted pyramidal Si devices consisted of the Si cathode, and an ~8 μ m thick film of a dry etchable polyimide having a dielectric constant $\varepsilon_r = 2.9$. The V-I characteristics for these devices exhibited high differential resistivities (~200 M Ω) and, not surprisingly, arrays larger than 6 × 6 could not be ignited. However,

introducing into the dielectric a thin film of a material having $\varepsilon_r > 5$ was found to have a dramatic impact on the stability of single devices and arrays. For example, Fig. 3 shows a photograph (left) and SEMs of a 30 × 30 array of 50 μ m square microdischarge device pixels.⁴ Comprising four 15 × 15 subarrays excited independently of one another, the array structure has an area of 8.5 mm² which corresponds to a device packing density >10⁴ cm⁻². Each microdischarge pixel has a multicomponent dielectric² consisting of ~0.9 μ m SiO₂, ~0.5 μ m Si₃N₄ and ~8 μ m of polyimide. A number of tests performed on these devices indicate that the silicon-nitride film is most responsible for the observed stabilization of the devices.

A photograph of the 30×30 array operating in 400 Torr of Ne is shown in Fig. 4. The large ignition voltage indicated on the figure (1130 V) is attributable to the resistivity of the Si substrate and does not, by any means, represent a lower fundamental limit. In fact, most arrays studied to date have operating voltages of only a few hundred volts. The right-hand portion of Fig. 4 is a photograph of the bright green fluorescence produced when a phosphor (Mn:Zn₂SiO₄), coated onto a thin sapphire substrate, is back-illuminated by a 30×30 array operating with a 500 Torr Ne/50 Torr Xe gas mixture. The green power radiated into a solid angle of $5 \cdot 10^{-2}$ sr was measured to be $14 \,\mu\text{W}$ and this emission is quite bright, even when viewed from a distance of several meters.

Further improvement in the spatial uniformity of the emission produced by these large arrays is clearly necessary (Fig. 4, left) and this subject is being pursued further under a subcontract to this laboratory from Ewing Technology Associates which received an SBIR Phase II contract from AFOSR. The goal of the SBIR work is to demonstrate arrays having 10⁴-10⁵ pixels and producing hundreds of mW in the UV. It should be emphasized, however, that the foundation of realizing large microdischarge arrays was laid with the support of AFOSR grant F49620-00-1-0372 and on the basis of our experience with these structures, we are confident that sealed, lightweight arrays driven at line voltage are feasible.

C. Ceramic Planar Arrays With Individually-Ballasted Pixels

In collaboration with the Motorola Solid State Technology Laboratory in Tempe, AZ, we have been studying microdischarge devices fabricated in multilayer ceramic structures. Ceramic is of interest not only because of its robust thermal and mechanical properties (and, hence, resistance to aggressive chemical environments) but also because of the enormous investment already made by industry in low-temperature co-fired ceramic (LTCC) technology.

Our initial microdischarge devices in ceramic multilayer structures⁵ had interleaving electrodes and microcavities that were drilled mechanically while the structure was still in the "green" (i.e., pre-fired) state. Although these devices did operate in rare gas pressures up to and

30 × 30 Microdischarge Arrays $(<9 \text{ mm}^2)$

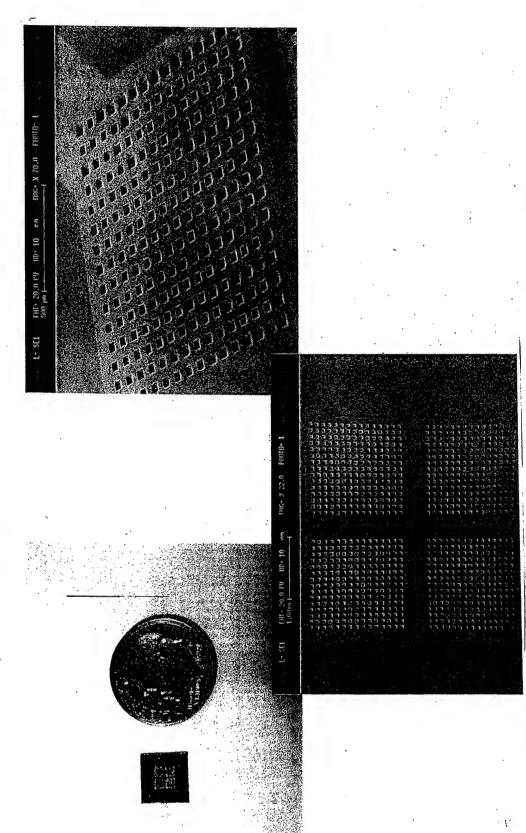


Fig. 3. Photograph (left) and SEMs of a 30×30 array of $(50 \ \mu \text{m})^2$ devices, consisting of four 15×15 subarrays driven independently.



Phosphor Excitation

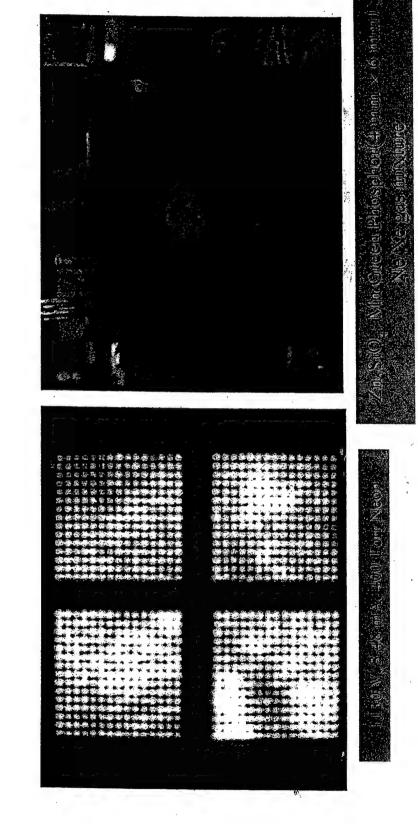


Fig. 4. Photographs of a 30×30 array operating in 400 Torr of Ne (left) and back-illuminating a phosphor (right), producing strong green emission.

beyond 1 atm, they were subject to intermittent failure, apparently because metal paste from which the electrodes are made was "smeared" along the discharge axis as the microcavity was machined. Also, the range in current over which the devices would operate was limited by the small cathode area.

A vastly superior design has been demonstrated recently that enables us to now fabricate large arrays in these structures. The key feature of the new design is that the electrodes are annular and recessed from the cylindrical microcavity to form a small chamber internal to the structure. The cathode surface area exposed to the plasma is five times larger than that in the early design, which has a profound impact on the device operating characteristics. Furthermore, the microcavity is punched in each layer of the structure *prior to* assembly. Finally (and, perhaps, most importantly) each pixel in an array is individually ballasted by a thick film (screen-printed) resistor integrated into the device structure.

The result is the realization of arrays that are among the most stable and long-lived that we have developed to date. The I-V characteristics for a 13×13 pixel array operating in Xe at pressures of 100-300 Torr are illustrated in Fig. 5. Notice that the devices are well-behaved and for the higher Xe pressures (200-300 Torr) and currents, the power delivered to the several cm² ceramic chip is >100 W! Not surprisingly, these structures produce intense emission. Figure 6 is a photograph of a 13×13 array of 200 μ m dia. ceramic devices operating in 300 Torr of Xe. The spatial uniformity of the emission is excellent and visual inspection of the arrays after several hours of continuous operation reveals no noticeable deterioration. It is already clear that this inexpensive technology offers an attractive route to manufacturing large area arrays:

D. Linear Ceramic Arrays: Platform for Microchip Lasers

The design of the planar arrays described in the last section has recently been adapted to produce linear arrays having active lengths >1 cm. Our motivation in pursuing this effort was to develop a structural platform suitable for exciting various gas and vapor lasers as well as serving as a photopumping source for microchip solid state lasers. The results have been gratifying.

In this design, the linear array consists of interdigitated Pt electrodes along its length. We find that displacing the anodes and cathodes along the axis of the microchannel improves the stability of the array dramatically. Each segment of the array is again individually ballasted by a thick film ruthenium oxide resistor. Figure 7 is a photograph of a seven segment linear array having an aperture of $80 \times 380 \ \mu\text{m}^2$ and an active length of ~1 cm. The array is operating in several hundred Torr of Ne and the visible emission produced by the array is sufficiently intense that it can readily be seen through the 0.5 mm thick upper layer of the ceramic structure.



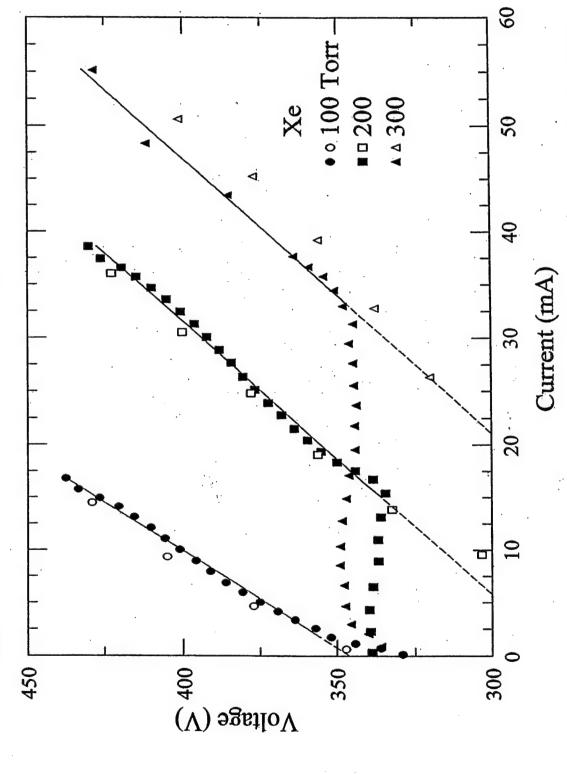


Fig. 5. I-V characteristics for a 13×13 array of 200 μm ceramic devices for three Xe pressures.

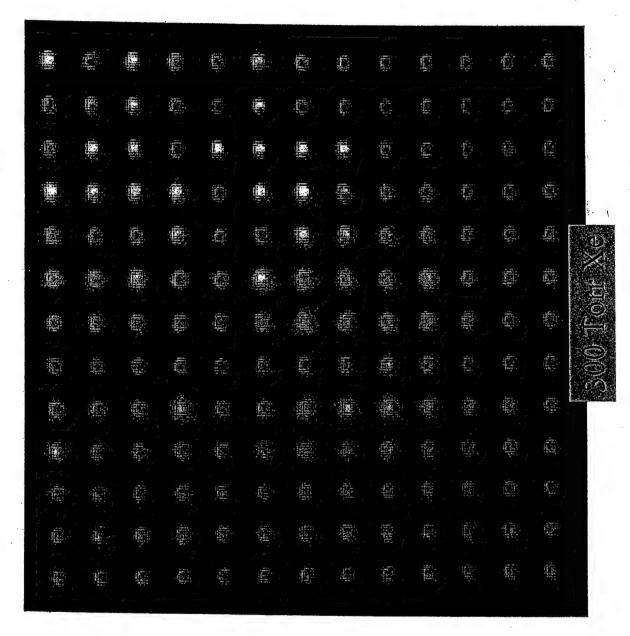


Fig. 6. Photograph of a 13 \times 13 array of 200 μm dia. devices operating in 300 Torr of Xe.

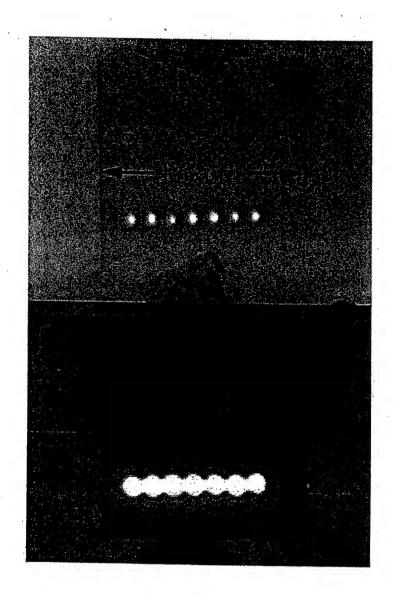


Fig. 7. Photograph of a seven segment linear array of microdischarges operating in Ne.

Experiments conducted with Xe at pressures ranging from 100 to 700 Torr suggest that the array produces optical gain on the 460.30 nm transition of the singly-charged Xe ion. Emission spectroscopy of several arrays having active lengths of ~1 mm to 1 cm show the 460 nm line intensity to increase rapidly with length and to do so more rapidly than the intensity of other ion transitions emanating from the same 4D_J manifold of Xe⁺. Although we have not yet installed a resonator around the device to produce an oscillator, we believe this result to be significant since it appears to represent the first microdischarge-pumped optical amplifier. It is interesting to note that the Xe⁺ 460.30 nm line originates from a state that is ~26 eV above the $5p^6$ (neutral) ground state and is one of the first rare gas ion transitions on which Bill Bridges demonstrated lasing in 1964!

We believe that the potential for this device structure as a platform for pumping various gas, vapor, or solid state lasers is great. These devices are extremely lightweight (0.5 g for a 1 cm active length structure), compact, and inexpensive. Section III outlines several proposed experiments with longer structures that we are eager to pursue.

E. Other Recent Results: Flexible Displays, Ceramic Dielectrics, 10 μm DRIE Devices

In addition to the accomplishments described above, several recent advances have been made, particularly with regard to flexible devices and arrays, which will be described briefly.

Figure 8 is a schematic diagram of a flexible microdischarge array we have demonstrated that is particularly promising for commercial production. The entire structure has a thickness of 30-40 μ m and is fabricated only by thick film (screen printing) and standard evaporation processes. Notice that each pixel in the array is individually ballasted by a film of ruthenium oxide. Polyimide is typically the dielectric and the anode and cathode are generally thin metal films. Also, the conducting substrate (metal foil or Kapton with a patterned metal film) is coated with a thin polyimide film to provide electrical isolation between the devices. The largest arrays fabricated and tested to date are 4×4 with $100 \ \mu$ m dia. microcavities. These arrays have excellent spatial uniformity, operate continuously at ~148 V and are extremely bright. We plan to submit a description of this work for publication in the near future and our next challenge is to find an inexpensive method for sealing the arrays.

Another flexible array concept that we have demonstrated successfully is printed onto Kapton. Electrode structures printed on both sides of the flexible Kapton substrate enable each pixel to be individually addressed. Film resistivity is, at the present time, limiting the uniformity of emission from the arrays but we are confident that we will be able to correct this problem with the proper choice of electrode material.

Thin Film Self-Ballasted Microdischarge Arrays

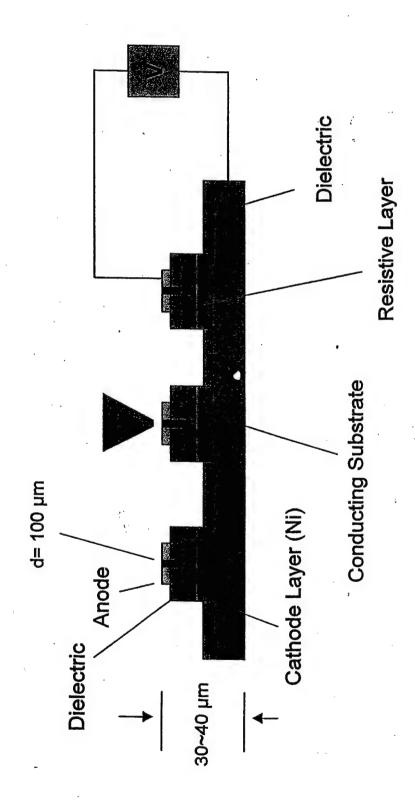


Fig. 8. Schematic diagram of a screen printed microdischarge array with individually ballasted

For applications requiring high temperature operation or contact with corrosive gases or vapors, we have developed a robust microdischarge architecture in which the dielectric is a film of a ceramic or other refractory material. Films of Al_2O_3 , BN, or $BaTiO_3$, ranging from 10 to 200 μ m in thickness, have been deposited on nickel foil by casting or printing processes. By eliminating all low temperature materials, we have demonstrated that these structures readily withstand continuous operation at temperatures of $1200^{\circ}C$. It is, perhaps, not surprising that these devices have outstanding lifetime characteristics. As illustrated in Fig. 9, the output power of a Ni screen/30 μ m BN/50 μ m Ni device operating in 700 Torr of Ne declines by only a few percent from its initial value after operating continuously for 100 hours. Inspection of the device after such tests shows no visible signs of deterioration and it appears that the only cause of the slight decrease in output power is degassing of the ceramic film, owing to the organic binder. Eliminating or minimizing this impurity should be straightforward with more attention given to the bakeout procedure. The Ni/ceramic/Ni device structure is so robust that we have operated these devices continuously in air (atmospheric pressure).

As a final example of the versatility of this technology, we have found that a small (three element) microdischarge array installed behind the cathode of an arc lamp will cut the ignition voltage for that lamp by at least a factor of two. We believe this to be a significant result for the lighting industry because lamp lifetime depends inversely (and nonlinearly!) on the ignition voltage. These results were published in *Applied Physics Letters* (ref. 6) and a patent application is pending.

F. Summary

This section has briefly described the considerable progress that has been made over the past 2.5 years under this AFOSR grant toward realizing the potential of microdischarge devices. Highlights include arrays that have been fabricated and tested in flexible (30-40 μ m thick), Si (30 × 30 pixels) and ceramic structures and individually-ballasted pixels have been produced by thick film (screen printing) processes, dramatically improving array emission uniformity. Perhaps the single most important result obtained thus far under this grant is the realization of a new, photodetector concept. This hybrid semiconductor/plasma device exhibits extraordinary sensitivity, approaching 10^3 A-W⁻¹, and appears to be quite general in operating principle, leading us to expect that this structure will perform equally well in the infrared and deep UV.

Lifetime

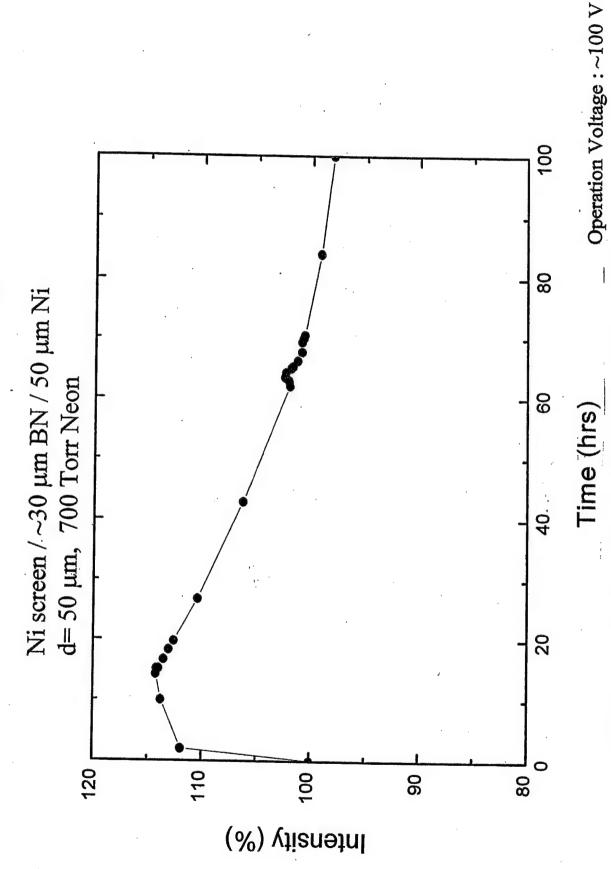


Fig. 9. Variation of the output intensity of a Ni/30 μm BN/Ni device (operating in 700 Torr of Ne) over a 100 h time period.

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III. PATENTS GRANTED UNDER AFOSR SUPPORT (2000-2003)

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